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BRIEF INTRODUCTION TO MIDAC AUTOQUANT™ VERSION 3.0

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13. ABSTRACT *(Maximum 200 words)*

This report documents the implementation of version 3.0 of the commercially available AutoQuant™ software package. Version 3.0 allows use of multiple alarms for individual compounds, temperature/pressure correction between sample/reference spectra, and a nonlinear analysis module to handle instrumental nonlinearities. This report discusses the procedures that are required for method development. This method development is reviewed for the target analyte ethanol. In addition, result discrepancies between processing raw interferograms in MDGrams 32 compared to AutoQuant™ are shown.

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PREFACE

The work described in this report was authorized under Contract No. DAAD05-97-P-3045. This work was started in August 1997 and completed in December 1997.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

At the time of this writing, Midac AutoQuant™ version 3.0 was still a beta release from Midac Corporation. There are likely to be some programming errors still present in the software. AeroSurvey, Inc., cannot be responsible for these errors. Likewise, AeroSurvey, Inc., has not rigorously tested Midac AutoQuant™ version 3.0 in a real-world monitoring situation and makes neither guarantees nor promises, implied or otherwise, about the performance of Midac AutoQuant™ version 3.0. AeroSurvey, Inc., will not be responsible for the application of Midac AutoQuant™ version 3.0 to FT-IR monitoring by anyone outside AeroSurvey, Inc.

The Midac AutoQuant™ version 3.0 software, as described herein, does not have sufficient commercial value to justify restricted distribution.

This report has been approved for public release. Registered users should request additional copies from the Defense Technical Information Center; unregistered users should direct such requests to the National Technical Information Service.

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BRIEF INTRODUCTION TO MIDAC AUTOQUANT™ VERSION 3.0

I. Introduction

I.A. The Purpose of this Manual

This manual is a supplement to "Hardware, Software, and Operational Manual for an Extractive FT-IR Air Monitoring System", a manual included as part of a training course that AeroSurvey, Inc. provided to the U.S. Army and U.S. Army contractors in November of 1997 at ERDEC in Aberdeen Proving Grounds, MD. This manual introduces the newest release of the software package Midac AutoQuant™. In no way is this manual intended to be a complete overview of Midac AutoQuant™ version 3.0. Rather, it is intended only as a brief introduction to the capabilities of the software.

I.B. An Introduction to Midac AutoQuant™ Version 3.0

Midac AutoQuant™ is a data collection and analysis package developed by Midac Corporation. AutoQuant™ provides both a hardware interface with Midac instrumentation and associated sampling equipment and a Classical Least Squares type algorithm for the analysis of FT-IR spectra.

The newest release of AutoQuant™ is version 3.0. This newest version of AutoQuant™ represents a marked improvement in versatility and applicability of AutoQuant™ to FT-IR monitoring. Figure I.B.1 shows the main menu of AutoQuant™ version 3.0. Some of the advantages that AutoQuant™ version 3.0 offers over older versions of AutoQuant™ include multiple alarms for individual compounds, control of peripheral hardware (valving, etc.), temperature and pressure correction between sample and reference spectra, and a nonlinear analysis module that uses multiple reference spectra to analyze situations that result in nonlinear response of the instrumentation.

Before continuing, an important note should be made concerning AutoQuant™ version 3.0. Specifically, there is a discrepancy between the results obtained from and MDGrams/32™ when spectra of the same sample are

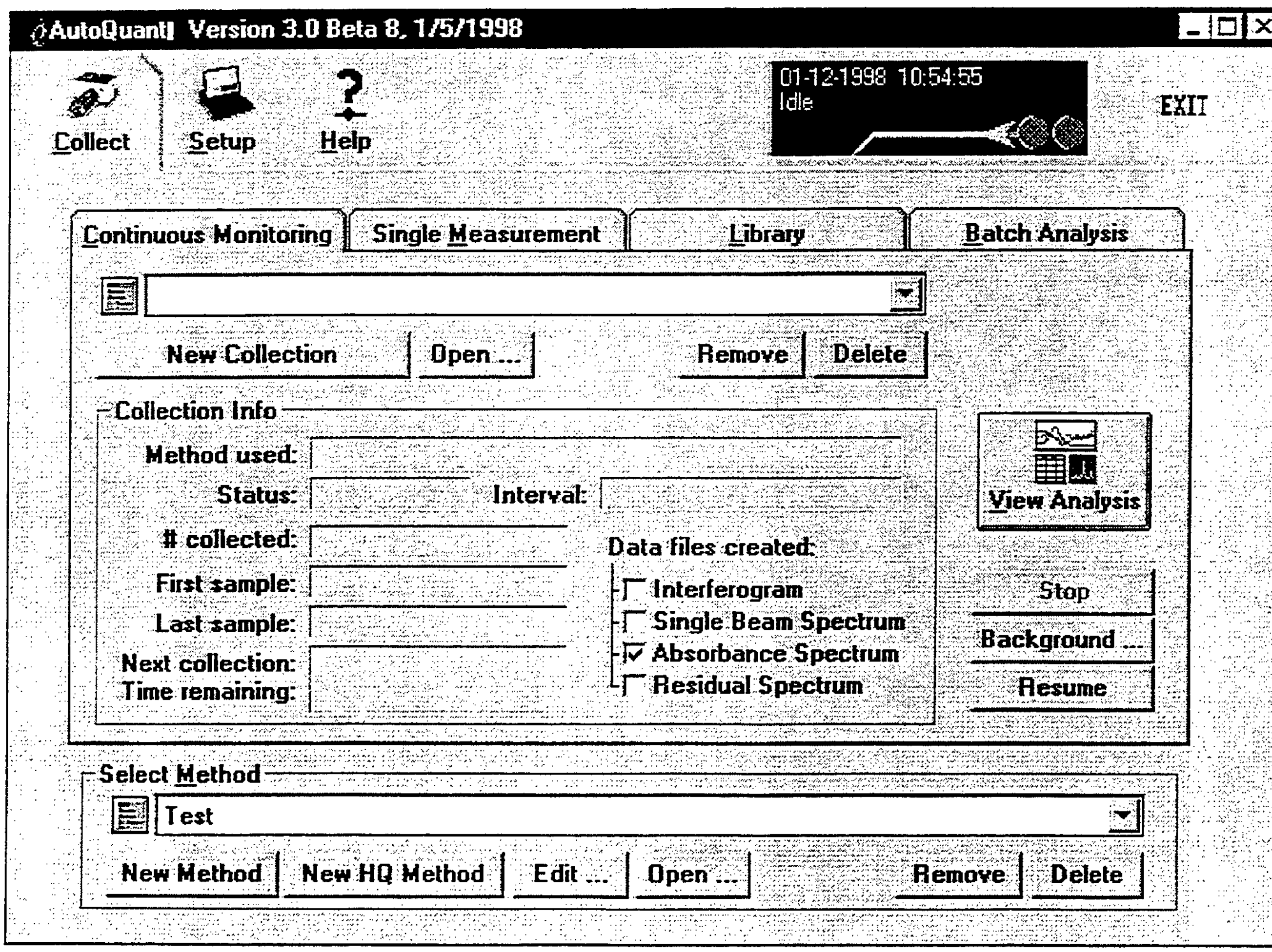

Figure I.B.1 The main menu screen of AutoQuant™ version 3.0.

collected and processed in each software package. This problem is discussed in more detail in Appendix A. The apparent discrepancy could result in grave quantitative errors if spectra from the two software packages are applied to one another in a quantitative analysis. Therefore, quantitative reference spectra and sample spectra must be collected and processed using the same software package and sampling protocols. See Appendix A for a more detailed discussion of this problem.

The core of all operations in AutoQuant™ is the analytical method that the user creates for a given experiment. This method determines how data is collected, processed, and analyzed by AutoQuant™. Once an analytical method is created, AutoQuant™ can be used to collect a single spectrum or perform continuous monitoring over an extended period of time. AutoQuant™ can also be used to collect and process reference library spectra or to analyze data that was collected and archived previously.

II Setting the Instrument Configuration and Experimental Parameters

II.A Checking the Instrument Configuration

Before data can be collected, the instrumentation being used must be configured to collect data in the proper manner. AutoQuant™ allows the user to check the instrument configuration from the Setup menu shown in Figure II.A.1 by clicking on the *Instrument Parameters* button. This terminology is slightly confusing. Most people would expect the *Instrument Parameters* button to lead to a menu where the spectral resolution, apodization function, gain, etc. could be set. However, the instrument parameters in the Setup menu in AutoQuant™ refer to the scan speed, and instrument/computer interface parameters to be employed by AutoQuant™. The normal instrument parameters (resolution etc.) are set in the analytical method and these will be covered in that section.

Figure II.A.2 shows the Instrument Configuration menu. Only one setting here that should be routinely changed. That setting is the Spectrum Format generated by

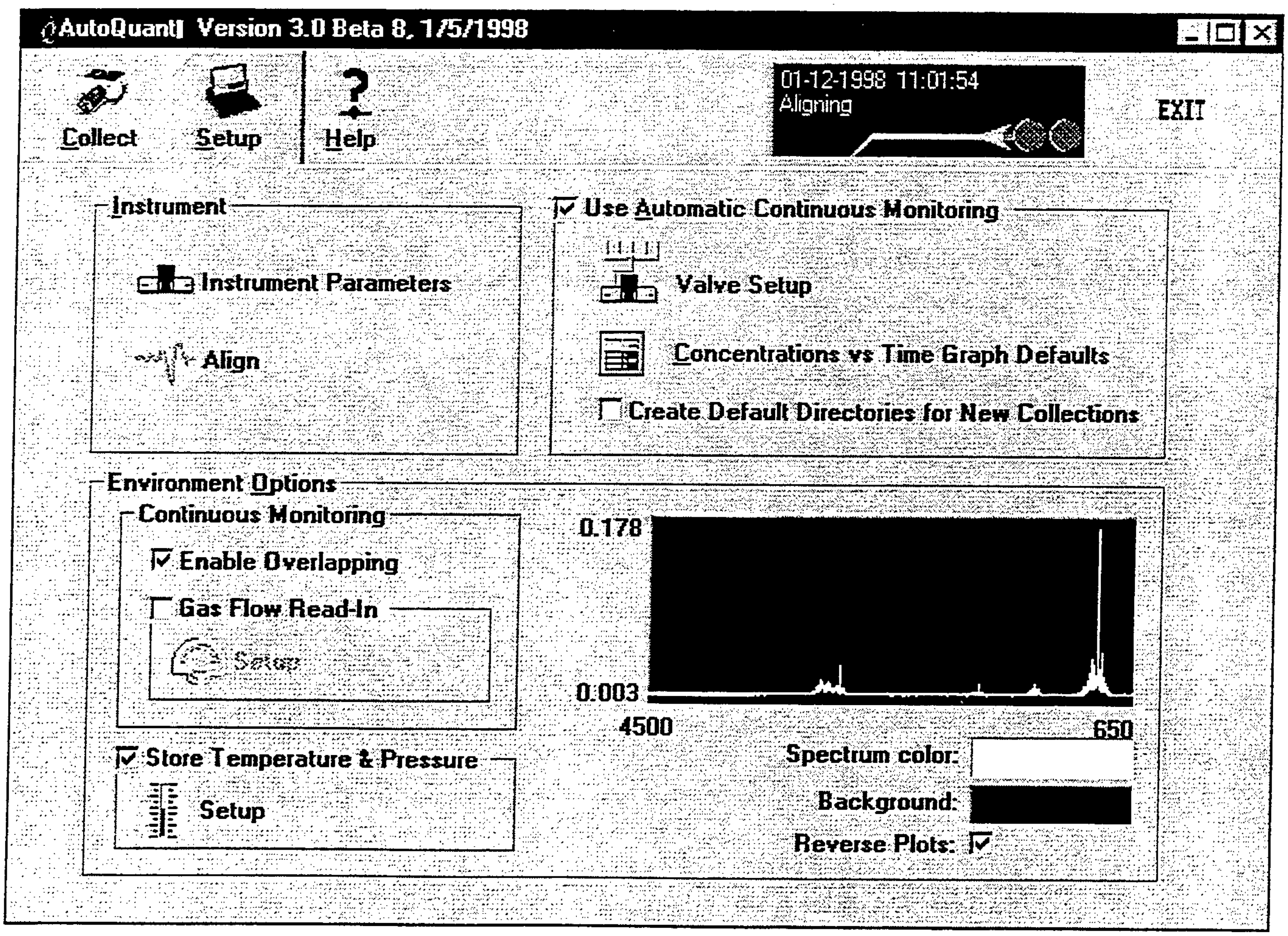

Figure II.A.1 The setup menu in AutoQuant™.

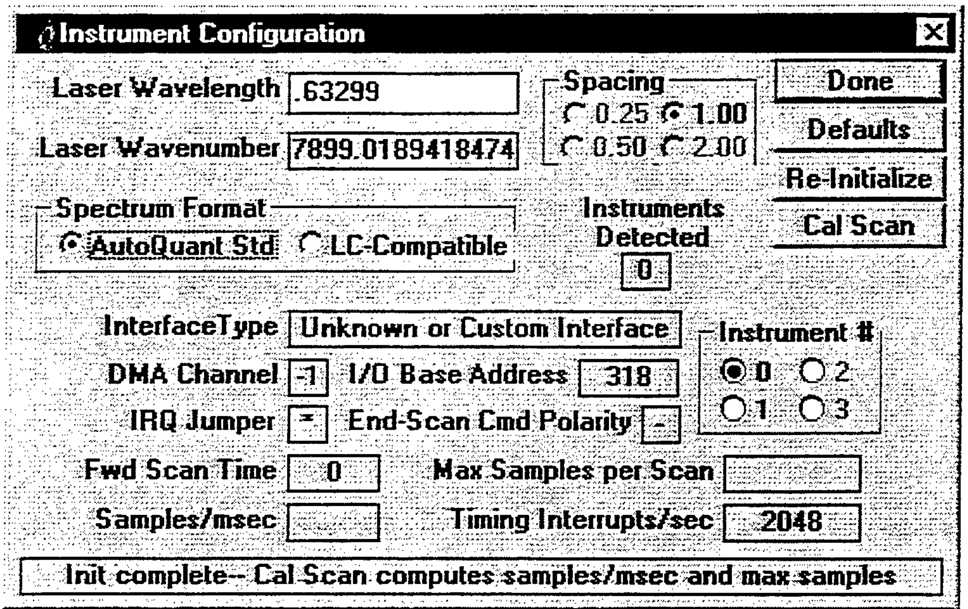

Figure II.A.2 The Instrument Configuration menu in AutoQuant™.

AutoQuant™. This Spectrum Format should be set to LC-Compatible files. This will allow the files collected in AutoQuant™ to be viewed and manipulated in MDGrams/32™ software from Galactic Industries. MDGrams/32™ is a more versatile spectral manipulation program and will afford the user greater flexibility in looking at spectra that are collected.

The remaining settings shown on the Instrument Configuration menu are generally to be left alone. The software will detect what settings are being used by the Midac instrument and use these in subsequent calculations. To change these values, one must change the settings at the instrumentation by manipulating jumpers on the hardware.

To verify that the instrument and computer are communicating and to check the alignment of the instrument and sampling system, the Align mode is used. Clicking the Align button enters this mode. Figure II.A.3 shows the Align menu. The user can set the number of data points to view, the number of colors to view, the gain and the spectral resolution to view while in align mode. Once these parameters are selected, the user can enter align mode by clicking the OK button. Once the Align mode is entered, the peak intensity and location of the ZPD of the interferogram can be seen. It is important that the positive and negative peak intensity of the interferogram be noted. These numbers tell the user how much of the ADC range is being used. The ADC peaks at 32768 counts and data should not be collected while the ADC

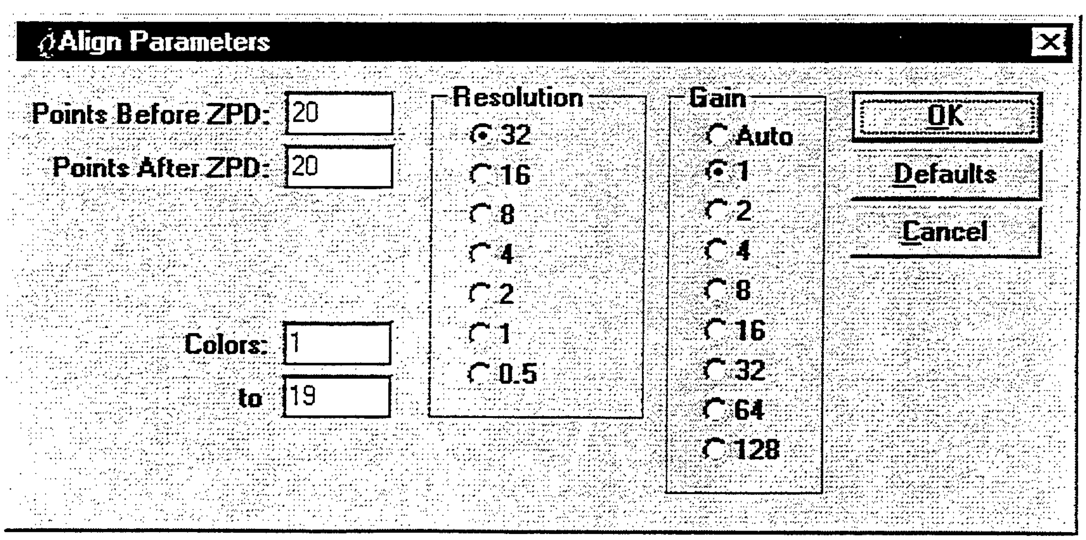

Figure II.A.3 The Align menu in AutoQuant™.

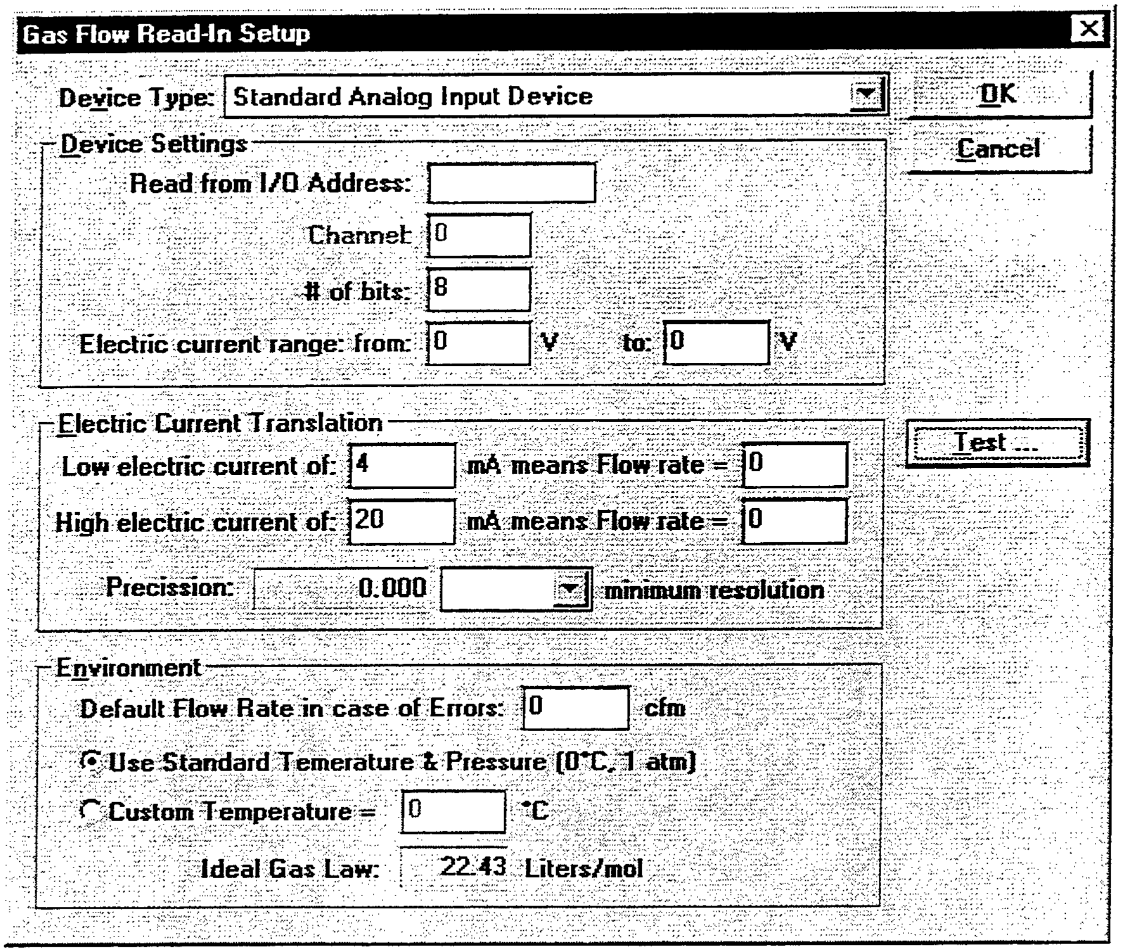

Figure II.B.1 The Gas Flow Read-In menu in AutoQuant™.

is peaked in either the positive or negative direction. Optimum numbers for the peak intensities of the interferogram are approximately 25000 counts.

II.B Configuring Peripheral Hardware

The peripheral hardware parameters that can be accessed from the AutoQuant™ Setup menu are external valving, temperature and pressure sensors, and mass flow sensors.

Figure II.B.1 shows the menu that can be used to setup an interface between AutoQuant™ and a mass flow controller that has an output of 4-20 milliamps. This interface will allow AutoQuant™ to directly read the flow rate of the sample system is in order to use this value in calculations such as total mass flux. External hardware that would be needed for this includes an appropriate mass flow controller, an analog I/O board in the host computer, and the appropriate cabling. Once communication is established with the mass flow controller, AutoQuant™ creates a file that logs the flow rate through the mass flow controller over time.

Figure II.B.2 shows the Temperature and pressure Access menu in AutoQuant™. This menu is used to communicate via an RS232 connection with a temperature and pressure sensor. Once communication with the temperature and pressure sensor is established, the relevant temperature and pressure for a given spectrum will be stored in the header of that spectrum as the spectrum is collected. AutoQuant™ will use this temperature and pressure data for subsequent calculations.

Figure II.B.3 shows the Valve Setup menu in AutoQuant™. This menu allows the user to control valving in the sample system for the purposes of purging the cell, running QA/QC samples or sampling multiple processes with the same instrument. In order to set up the valve control, TTL I/O board(s) must be installed in the host computer. This board or boards must have enough channels to handle all of the valving required for the experiment. By controlling the system valving in AutoQuant™ the sample system can be purged and QA/QC samples can be ran throughout the experiment without actually having to stop the collection in AutoQuant™. This setup would require the appropriate TTL I/O board or boards, the proper automatic valving and the appropriate cabling.

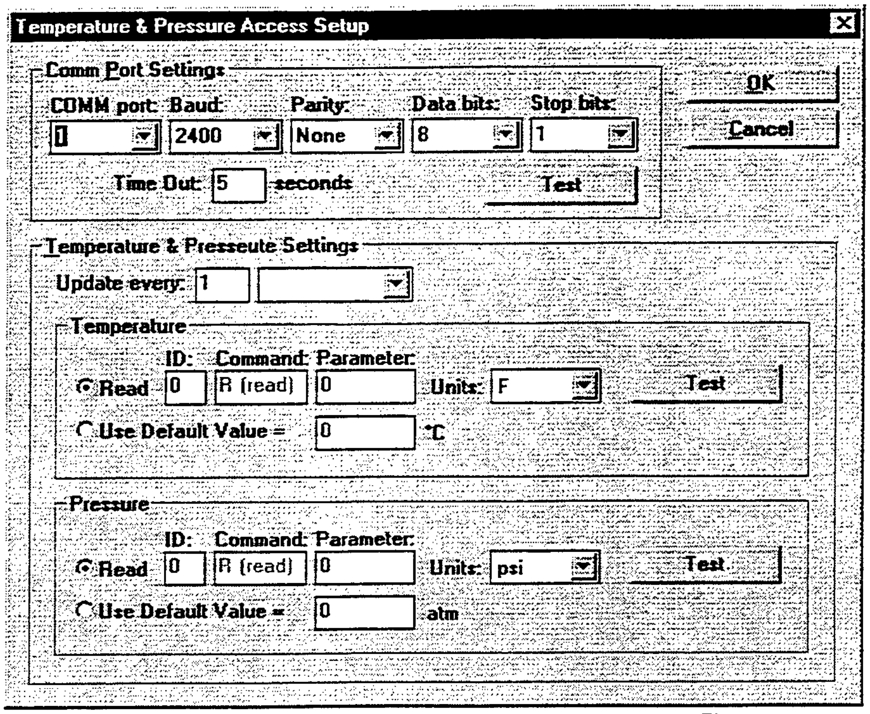

Figure II.B.2 The Temperature and Pressure Access menu in AutoQuant™

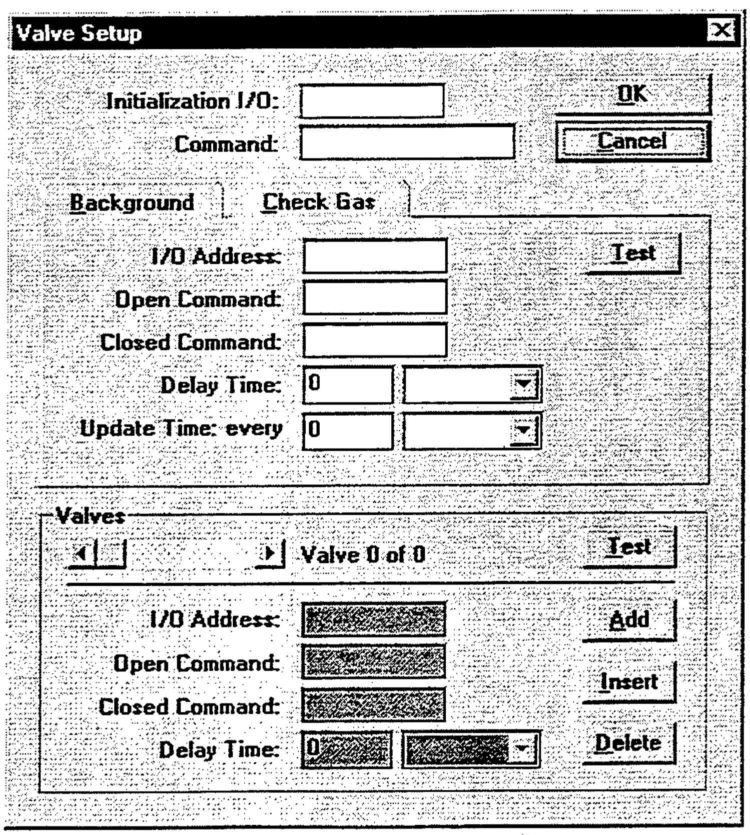

Figure II.B.3 The Valve Setup menu in AutoQuant™.

II.C Configuring the Concentration vs. Time Graph

A graph of concentration versus time is the visual output of AutoQuant™ after an analysis has been performed. This output can be viewed in real-time during a continuous collection or generated during a batch analysis. The actual numeric concentration values can be saved as a text file for import into a spreadsheet or database program.

The concentration vs. time graph is straightforward as can be seen in Figure II.C.1. The units in which the analyte levels will be reported (y-axis units) must be set first. The choices are either in a concentration format (i.e., part per million) or a mass emissions format (i.e., pounds per hour). In order to report mass emissions the sample flow rate must be measured and input into AutoQuant™. The user can have the error bars calculated by AutoQuant™ shown on the graph or can omit them if the graph view appears too cluttered.

After the y-axis units are determined, the user must determine the x-axis (time) units. The choices here are seconds, minutes, hours, days, or an actual 12 or 24 hour clock.

The user can also choose how many samples should be displayed. All samples or some set number of samples can be displayed, or the most recent samples collected over some given amount of time can be displayed.

III Collecting Library Spectra

As mentioned earlier, there is a discrepancy between the results obtained from AutoQuant™ and MDGrams/32™ when spectra are collected and processed in each software package. This discrepancy could result in grave quantitative errors. **Therefore, quantitative reference spectra and sample spectra must be collected and processed under the exact same conditions and using the same software package.** See Appendix A for a more detailed discussion of this problem.

Creation of a Reference Spectrum Library

The analytical process starts with the collection of high quality reference spectra. AutoQuant™ affords the user the ability to collect reference spectra and create different libraries for different monitoring tasks. Figure III.A.1 shows the main Library Spectrum Collection menu.

If the reference library spectra to be collected are for a new library, then the *New Group* button should be selected. This option presents the user with the menu shown in Figure III.A.2. This menu allows the user to name the library that is going to be created and assign a subdirectory to the library on the host computer. Alternatively, if spectra are to be added to an existing library, the appropriate library can simply be selected on the main Library Spectrum Collection menu.

The first step to be taken in generating a reference library spectrum is to collect a background spectrum. The background spectrum should be collected under the exact same operating conditions that will be used for the collection of the sample reference spectrum. Figure III.A.3 is the Library - New Background menu in AutoQuant™. This menu allows the user to enter a description of the file being collected, name the file, and select the spectral resolution and the number of coadded scans to be used when collecting the spectrum. Other instrument parameters such as gain, spectral

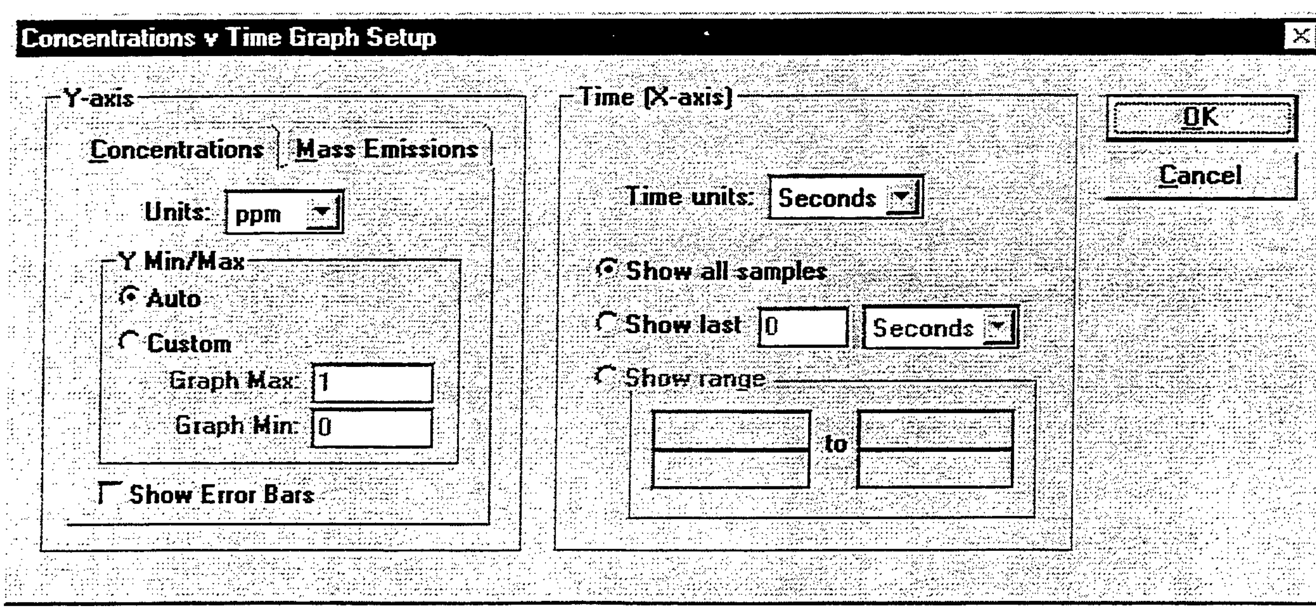

Figure II.C.1 Concentration versus Time Graph Setup menu in AutoQuant™

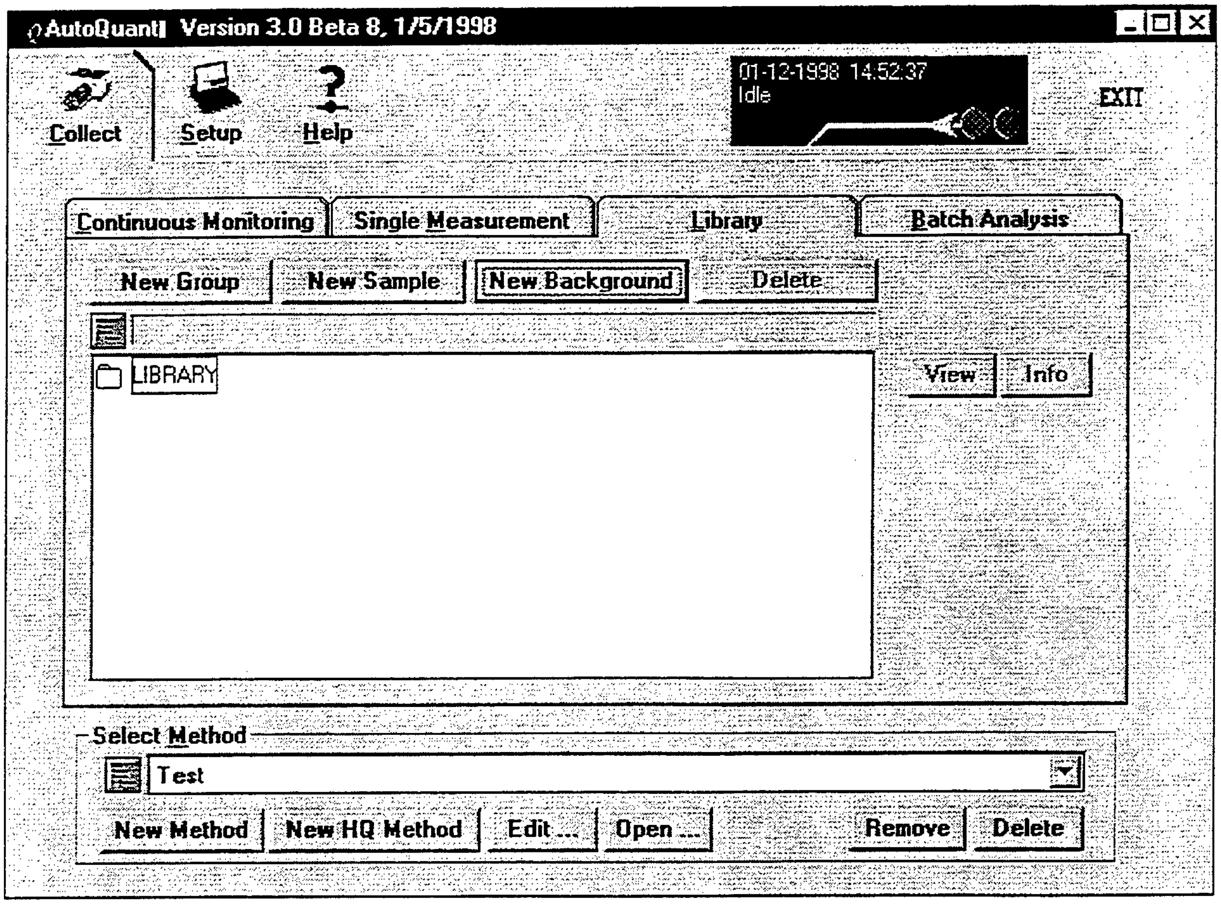

Figure III.A.1 The main view of the Library Spectrum Collection menu in AutoQuant™.

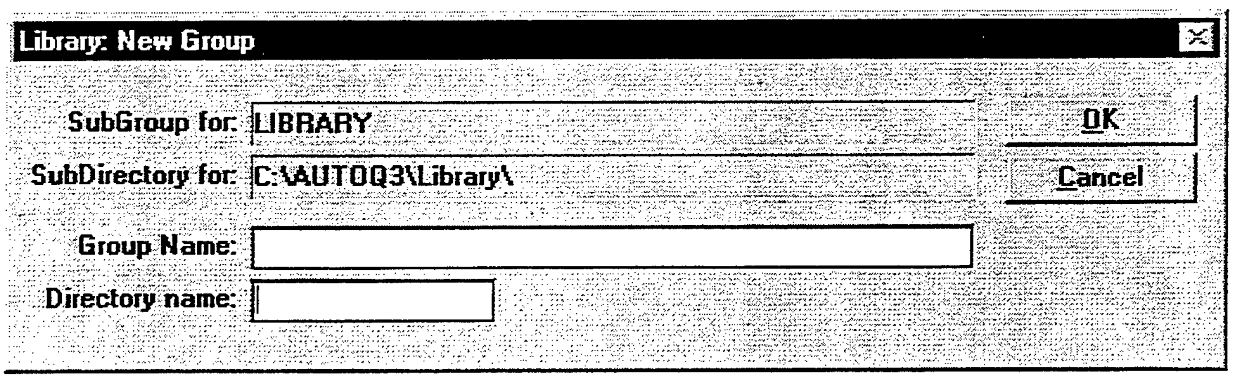

Figure III.A.2 The Library New Group menu in AutoQuant™.

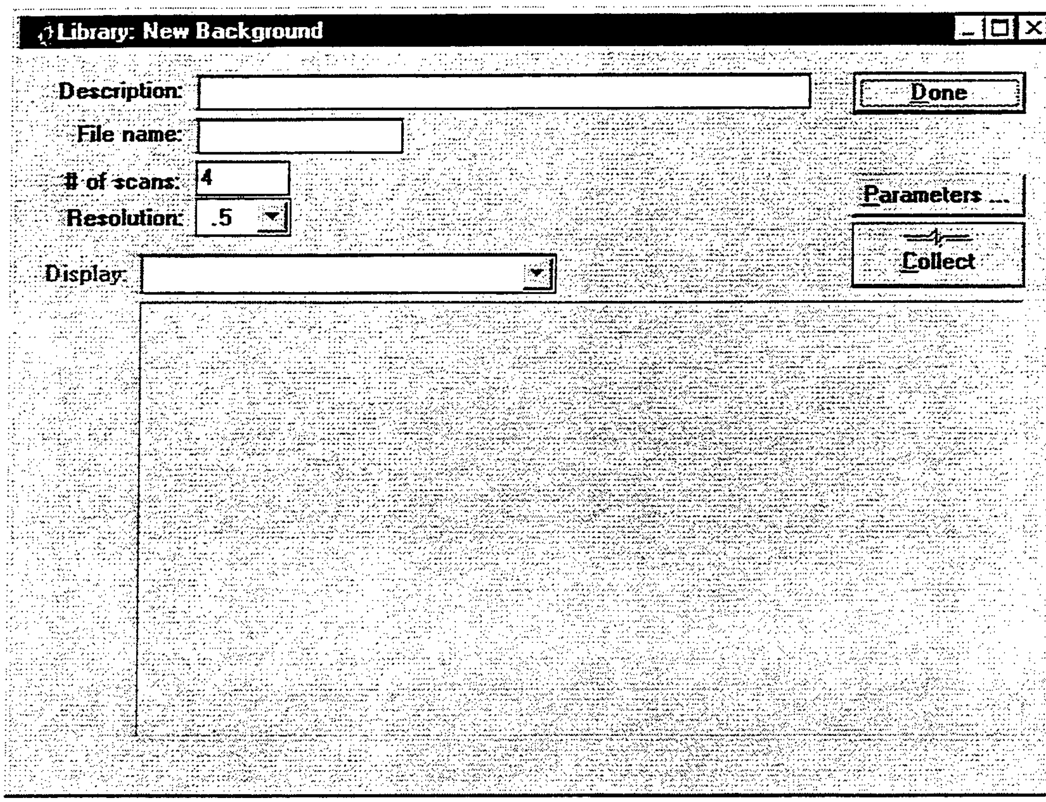

Figure III.A.3 The Library - New Background menu in AutoQuant™

range, and apodization function can be set by clicking on the *Parameters* button and entering the Collection Parameters menu. The Collection Parameters menu is shown in Figure III.A.4. Again, it is very important that the sample and background spectra used to create a reference library spectrum be collected using identical conditions and instrumental parameters. The only difference between the two should be the introduction of the sample of interest.

Once all of the instrument parameters are set, the background spectrum collection is initiated by clicking the *Collect* button. When the spectrum has been collected, the single beam spectrum will be displayed in the bottom viewer of the Library - New Background menu.

Once a background spectrum is collected, a sample reference spectrum can be collected. This is done using the Library - New Sample menu shown in Figure III.A.5. If the

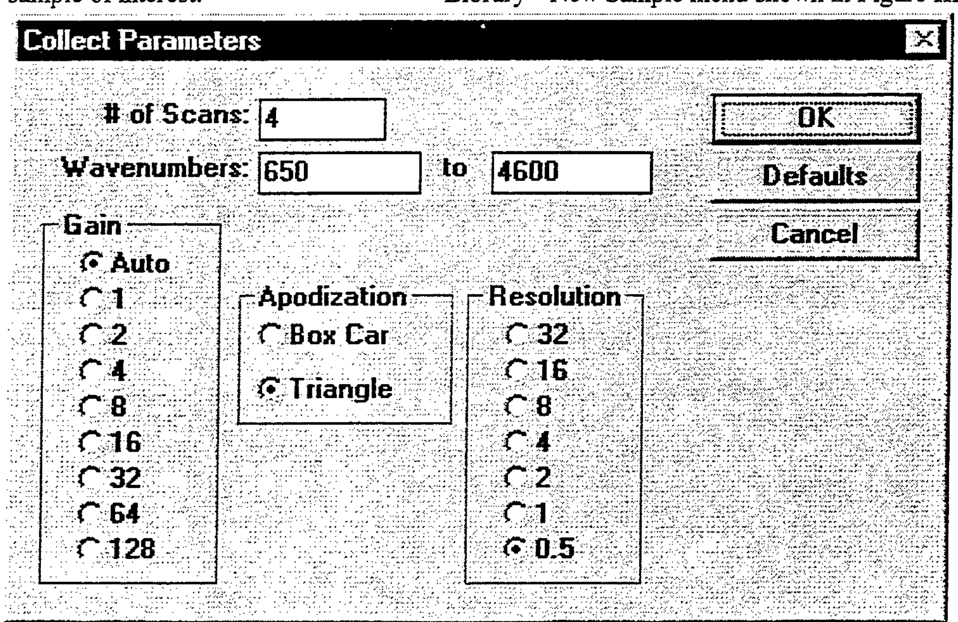

Figure III.A.4 The Collection Parameters menu in AutoQuant™

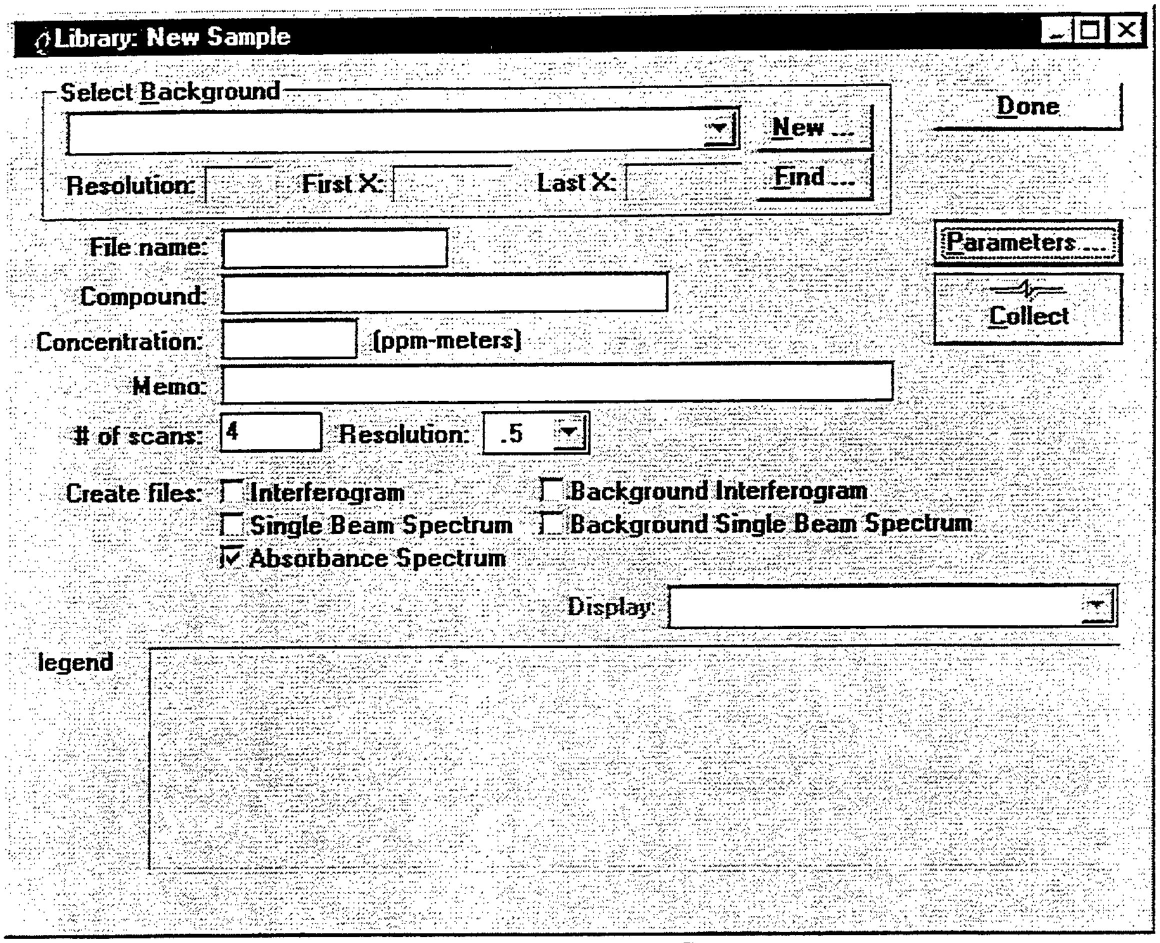

Figure III.A.5 The Library - New Sample menu in AutoQuant™

new background was just collected, this background should be displayed in the *Select Background* portion of the menu. If an old background spectrum is to be used, it can be selected by clicking the *Find* button. If a new background needs to be collected, the Library - New Background menu can be reached by clicking the *New* button.

After the background is selected, the pertinent information about the sample reference spectrum to be collected should be entered. The Collection Parameters menu should be reviewed on Collection Parameters menu (click *Parameters*) to ensure that these parameters match the background file. Next, the files to be generated should be checked. The choices here are to collect and save the interferogram, sample single beam spectrum, and/or sample absorbance spectrum.

Once these choices are made, the sample reference spectrum collection is initiated by clicking the *Collect* button. After the spectrum is collected, the files chosen to be saved will be written to the disk of the host computer and the spectra will be displayed on the bottom viewer of the Library - New Sample menu.

IV. Creating an Analytical Method

The analytical method is the heart of the data collection and analysis performed by AutoQuant™. This method determines the collection parameters and collection frequency during a continuous collection. The analytical method also determines the reference spectra used to perform quantitative analysis of the sample spectra collected during a monitoring project. Therefore, all results obtained from AutoQuant™ depend upon the creation of a proper analytical method.

Creating an Analytical Method

From the main menu of AutoQuant™, a user can access existing methods or create a new one. In order to access an existing method, the user need only click on the pull down menu in the Select Method portion of the main menu of AutoQuant™, Figure I.B.1. This will provide a list of methods that have been recently accessed or created. If the method that the user needs is not on the pull down menu, the user can click the *Open* button and browse the disk drive of the host computer for the desired menu.

If the method to be used does not exist, one can be created using the method editor. Access the method editor for the purpose of creating a new method by clicking on the *New*

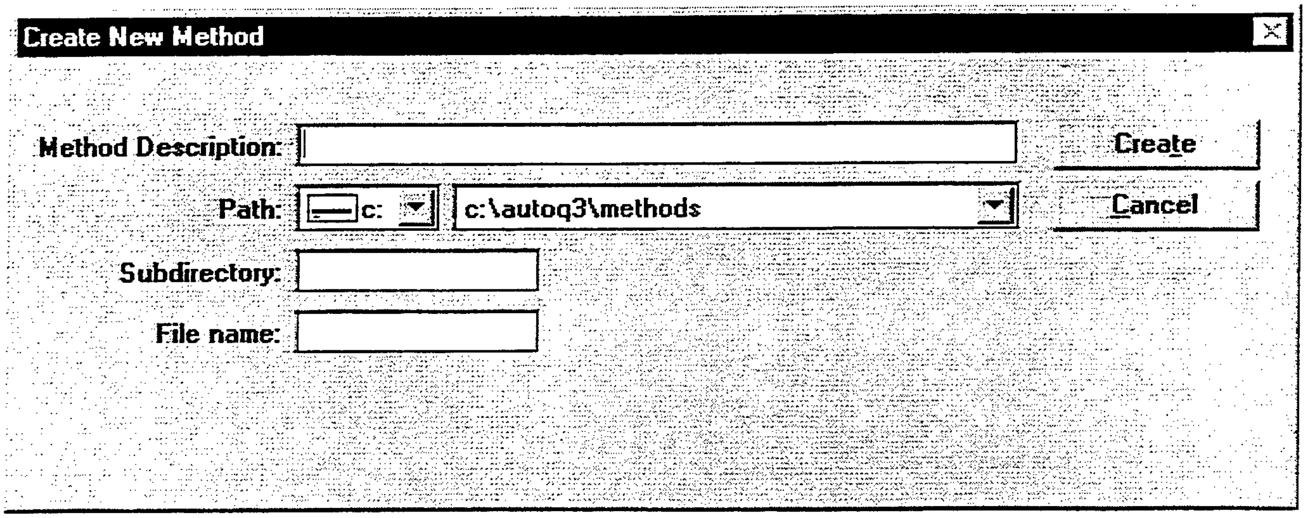

Figure IV.A.1 The Create New Method menu in AutoQuant™.

Method button on the main menu. The first menu that appears is shown in Figure IV.A.1 and is titled the Create New Method menu. This menu allows the user to type a meaningful description of the method, name the method and assign the method to a subdirectory on the host computer. Once these tasks are done, the user can click on the *Create* button, causing the Method Editor shown in Figure IV.A.2 to be displayed.

In the method editor, the first choice that needs to be made is whether the analysis is linear or nonlinear. A linear analysis should be used for monitoring situations that are well behaved (i.e. always follow Beer's law). This type of analysis employs one reference library spectrum for each compound to be analyzed and uses only one sub-method to perform the analysis.

A nonlinear analysis should be employed when the user feels there is a good chance that the process being monitored will show excursions from Beer's law. In a nonlinear analysis, multiple reference spectra are used for each compound to be analyzed. A sub-method is built for

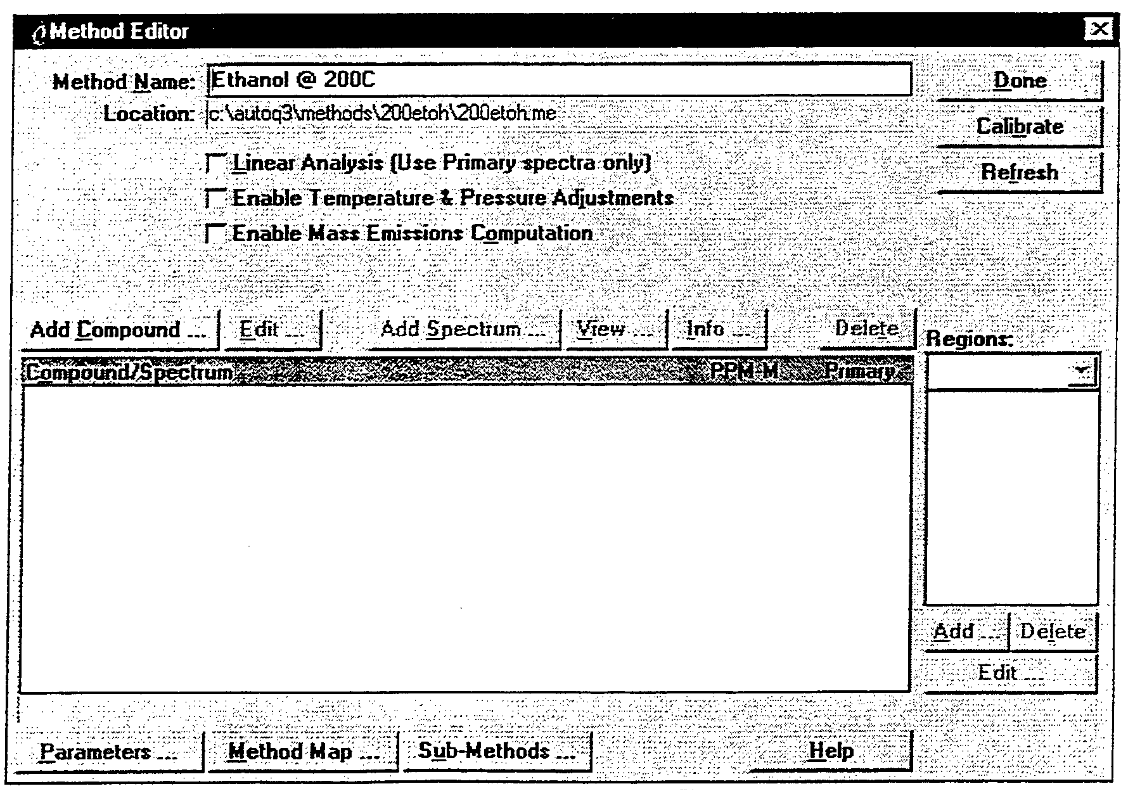

Figure IV.A.2 The main menu of the Method Editor in AutoQuant™.

each possible combination of reference library spectra used in the method and the results that are reported are taken from the sub-method that most closely resembles the sample. In other words, the sample is analyzed by all sub-methods, the final results that are reported are taken from the analysis of the sample by the sub-method that has analyte concentrations that most closely match the analytical results of the sample analysis.

The example method created for the purposes of this document will employ only a linear analysis.

Next, the user should decide whether AutoQuant™ should make corrections for temperature and pressure during the analysis. During a continuous collection, this correction requires that the software be interfaced with a temperature and pressure sensor. If performing a Batch analysis, the temperature and pressure of the sample can be specified, but must remain constant throughout the analysis.

The user also needs to specify whether or not AutoQuant™ should perform mass emission calculations. Similar to the temperature and pressure calculations, the software must be interfaced with a mass flow meter in order to perform these calculations during a continuous collection. The flow rate can be specified for a batch analysis but must remain constant for the entire analysis.

Once these decisions are made, the user may begin to add compounds to the method by clicking the *Add Compound* button. Figure IV.A.3 shows the Add Compound menu of the Method Editor. It is here that the temperature, pressure and mass of the compound must be specified if AutoQuant™ is to calculate any values relating to these parameters. Also, it is here that compound specific alarms can be set to warn users if the concentration of a compound exceeds or falls below some set value. Finally an analog output for the compound concentration can be set on this menu.

There are four compound specific alarms available in AutoQuant™. Highlighting the Trigger Alarms box and clicking on the Setup button configures these alarms. Figure IV.A.4 shows the Edit Compound Alarms menu. There are two "high alarms" and two "low alarms" that can be configured for each compound. The low alarms will trigger if the concentration of the compound falls below the set amount while the high alarms will trigger if the concentration of the compound exceeds the set concentration. Each alarm can be tied to an external device through a TTL I/O card in the host computer. The I/O card can then be made to trigger an external alarm device (i.e. a light).

The ability to output the concentration of a given compound to an external device requires an analog I/O card in

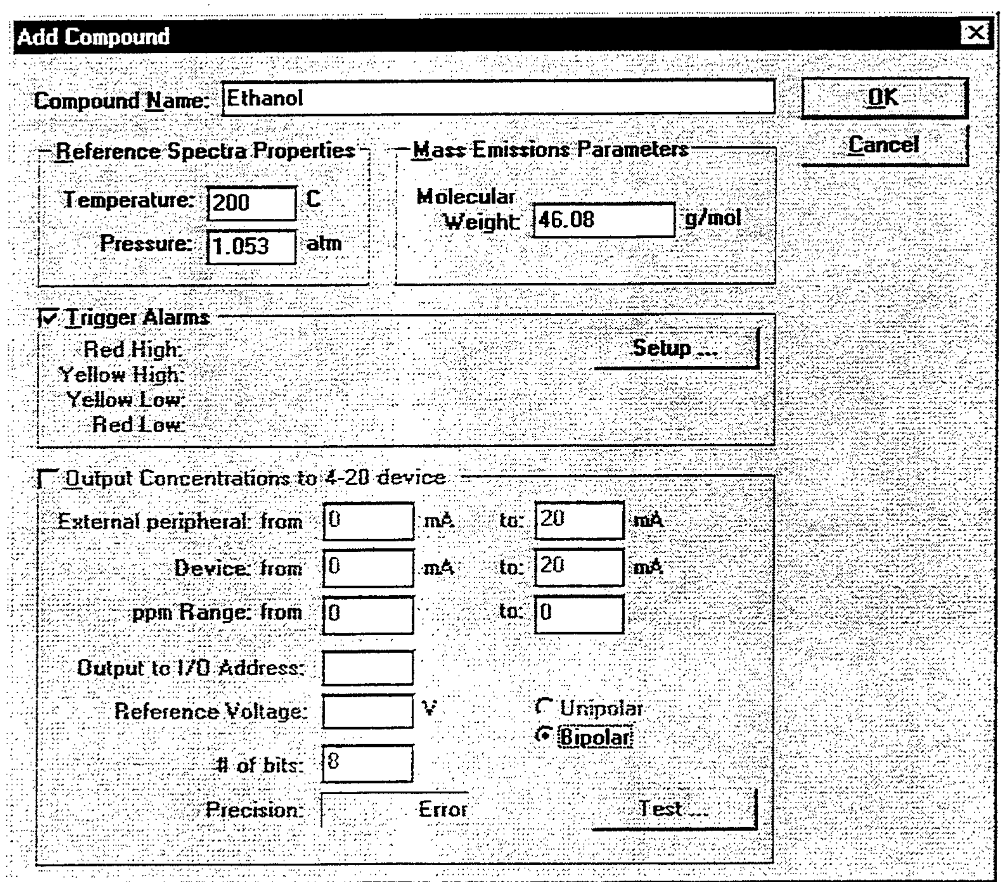

Figure IV.A.3 The Add Compound menu in the Method Editor of AutoQuant™

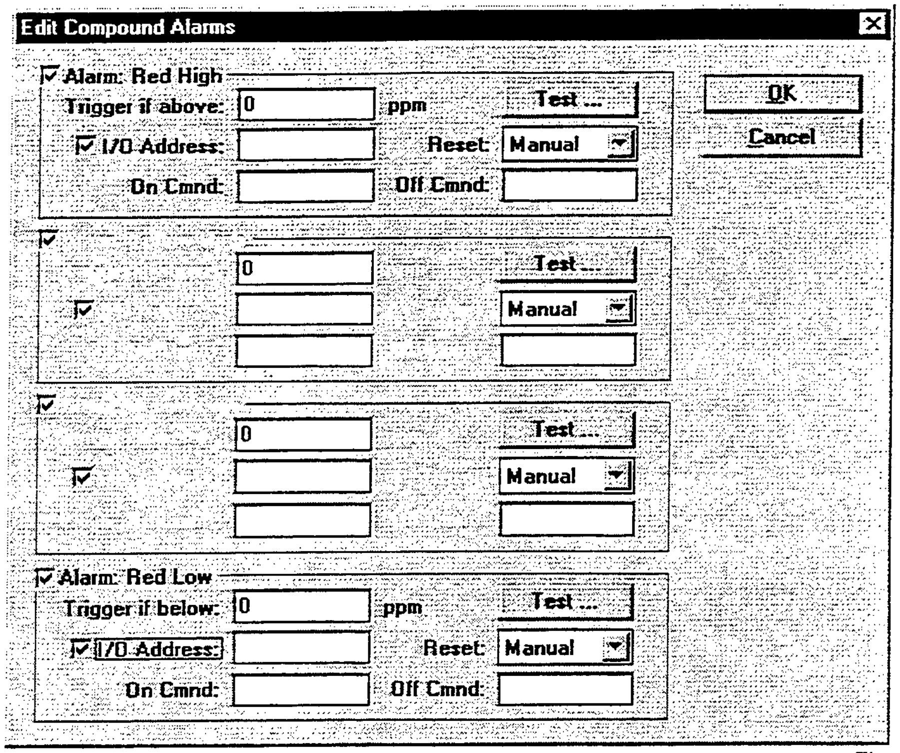

Figure IV.A.4 The Edit Compound Alarms menu in the Method editor of AutoQuant™.

the host computer, a recording device, and the proper cabling. This function can be used to create a strip chart recording of the concentration of a given compound for archiving purposes.

Once all of the pertinent information for the compound is entered, the user can return to the Method Editor and begin to add reference spectra to the method for each compound. As mentioned earlier, only one spectrum is used per compound for a linear analysis, but multiple spectra (preferably covering the full range of expected sample concentrations) are used for a nonlinear analysis.

In order to add a spectrum to the method, highlight the compound name in the Compound/Spectrum box of the Method Editor and click the *Add Spectrum* button. The window shown in Figure IV.A.5 will be displayed. The hard

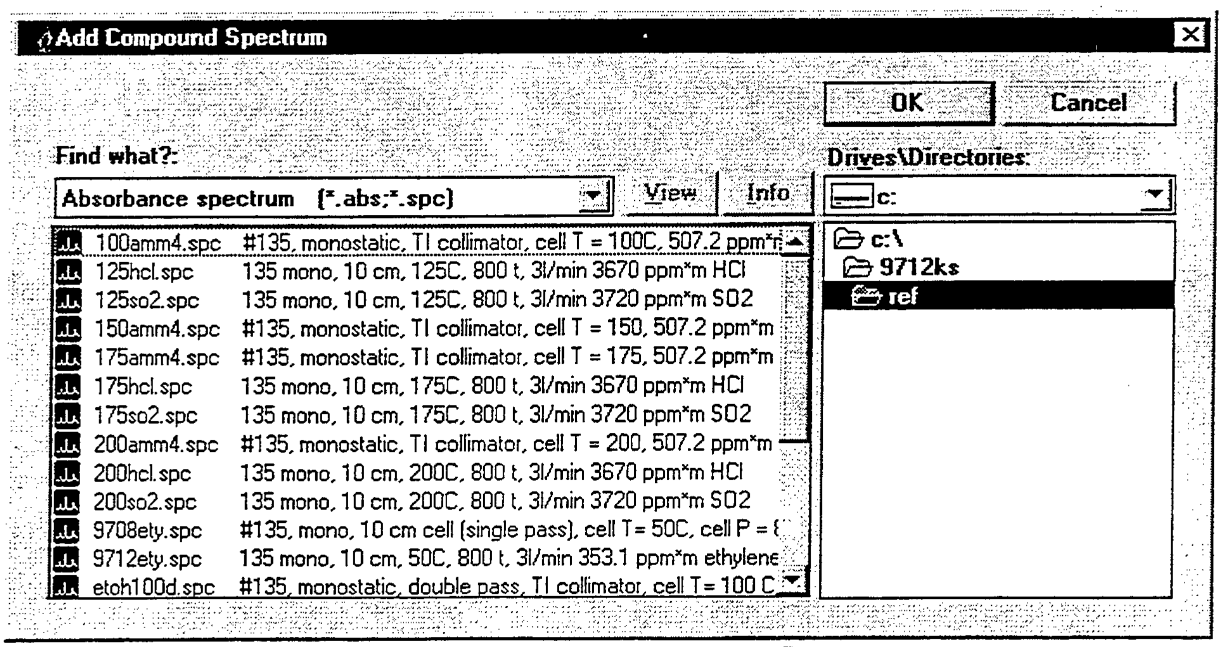

Figure IV.A.5 The Add Compound Spectrum window in AutoQuant™.

drive of the host computer can be browsed to locate the desired spectrum. Double-click on the desired spectrum's filename and that spectrum will be added to the method. Once a spectrum is added to the method, the concentration is automatically read from the header or memo of the spectral file. The user should double-check that this concentration is correct. If the concentration is not correct, double-click on the concentration and enter the proper concentration for that reference spectrum.

The final step of adding a spectrum to the method is to add analytical regions for each spectrum. The analytical regions are the regions over which AutoQuant™ will analyze the sample spectrum for the presence of the reference spectrum. Multiple regions can be used for one compound and any region that contains information about the compound should be used for the analysis. An exception to this rule may be regions of the spectrum that have large interferences from atmospheric or other compounds. Clicking on the *Add* button at the bottom of the Regions window in the Method Editor allows the analytical regions to be specified. The Method Editor is show in Figure IV.A.6 after compounds and spectra have been added to the method and the analytical regions for the method have been specified.

After the analytical regions are added to the method, the parameters should be set for the method. The Method Parameters menu is accessed by clicking on the *Parameters* button at the bottom of the Method Editor. Figure IV.A.7 shows the Method Parameters menu. These parameters will be used to collect and analyze data with this method. The data collection parameters set here should match, as close as possible, the parameters used to collect the reference library spectra that are being used by the method. The processing parameters that are selected here should be those that minimize the error of the results. In order to determine these parameters, the method should be tested on sample spectra using different processing parameters and the parameters that minimize the resulting error should be used for the final method.

Once the method parameters are set, the method should be calibrated. The process of calibrating the method simply performs some matrix manipulations that will allow AutoQuant™ to complete the calculations more quickly during the actual data analysis.

V. Collecting a Single Measurement

Once an analytical method is developed it can be applied to the measurement of sample spectra either singly or in a continuous monitoring experiment. This section will describe the application of the analytical method to an individual measurement

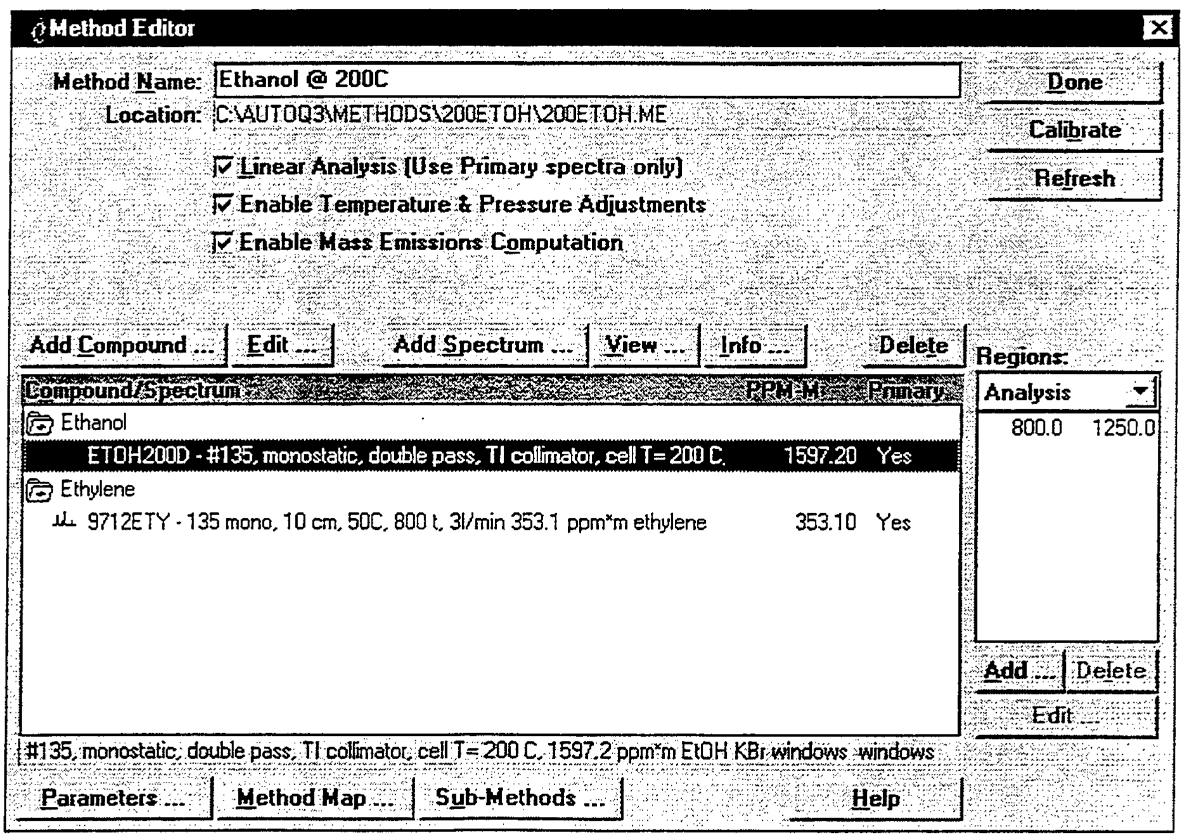

Figure IV.A.6 The Method Editor in AutoQuant™ after the addition of compounds and spectra.

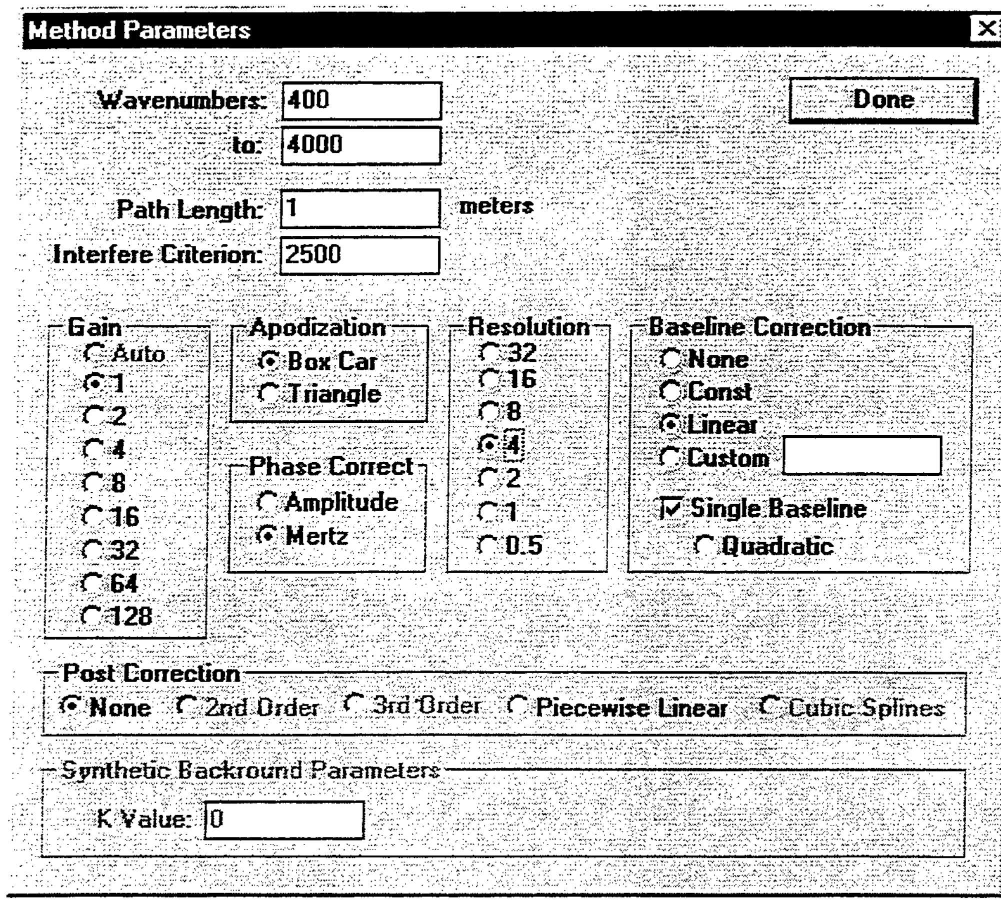

Figure IV.A.7 The Method Parameters menu in AutoQuant™.

V.A. Collecting a Single Measurement

There are times when only a single measurement of a sample is required. For those times, AutoQuant™ has the ability to make a single measurement of a sample. Figure V. A.1 shows the Single Measurement menu in AutoQuant™. This menu allows the user to collect individual spectra of a sample. The collection parameters are set by the analytical method that is selected at the bottom of the menu in the Select Method area. Individual spectra will be collected, processed and analyzed by the parameters set in this method.

In order to begin a single measurement, click on the *New Measurement* button on the Single Measurement menu. This opens the Single Point Measurement menu that is shown in Figure V.A.2. This menu allows the user to select or collect a background spectrum, name the sample file, enter a meaningful description of the measurement, and specify the compound being sampled and the expected concentration. The number of coadded scans can also be specified on this menu.

Once the pertinent information is entered, the measurement is initiated by clicking on the *Collect* button. AutoQuant™ will then collect, process and analyze the sample spectrum according to the analytical method. The results of the collection and analysis are displayed on the main Single Measurement menu.

VI. Performing a Continuous Monitoring Experiment

Long term continuous monitoring of processes is the application for which AutoQuant™ was truly designed. In these applications, AutoQuant™ allows the user the to select the frequency of samples, set alarms to notify personnel of potential problems, control both FT-IR instrumentation and peripheral hardware, and view analysis results in real-time.

Performing a Continuous Monitoring Experiment

Figure VI.A.1 shows the main menu for Continuous Monitoring applications of AutoQuant™. This menu allows the user to select a previous collection and view the analysis results or to create a new collection. To view a previous collection, click on the pull down menu at the top of the main menu. If the collection of interest is present in the list, double-click on the name. If the collection of interest is not present in the list, click on the *Open* button and browse the hard drive

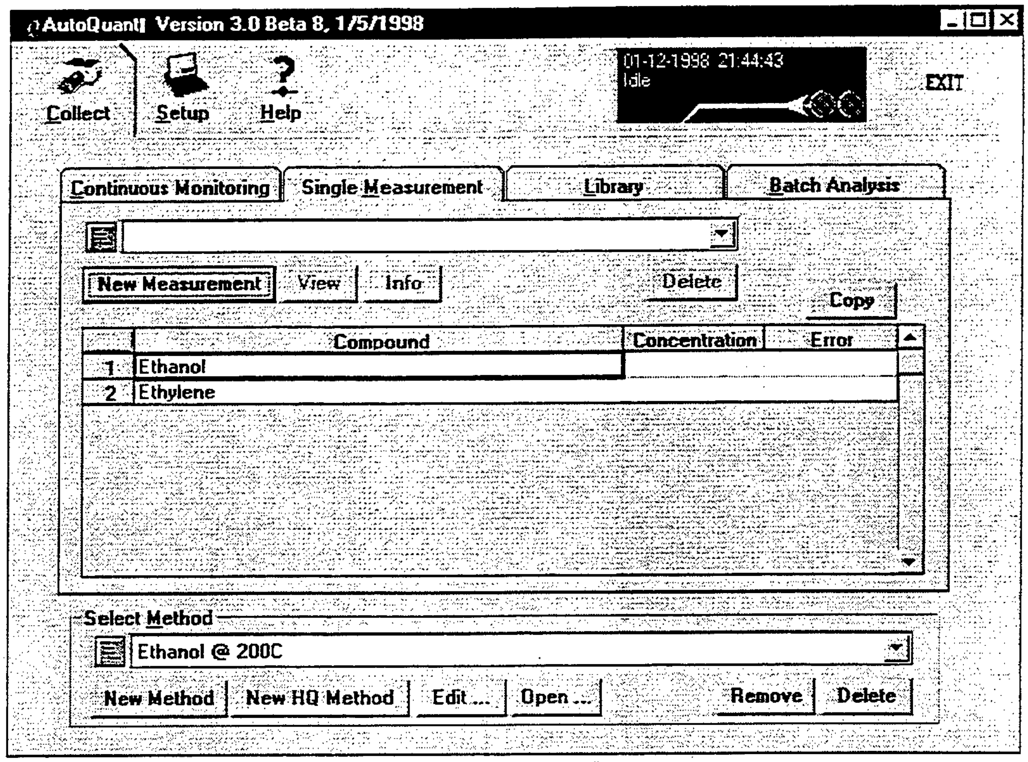

Figure V. A.1 The Single Measurement menu in AutoQuant™.

of the host computer. Once a collection is active on this

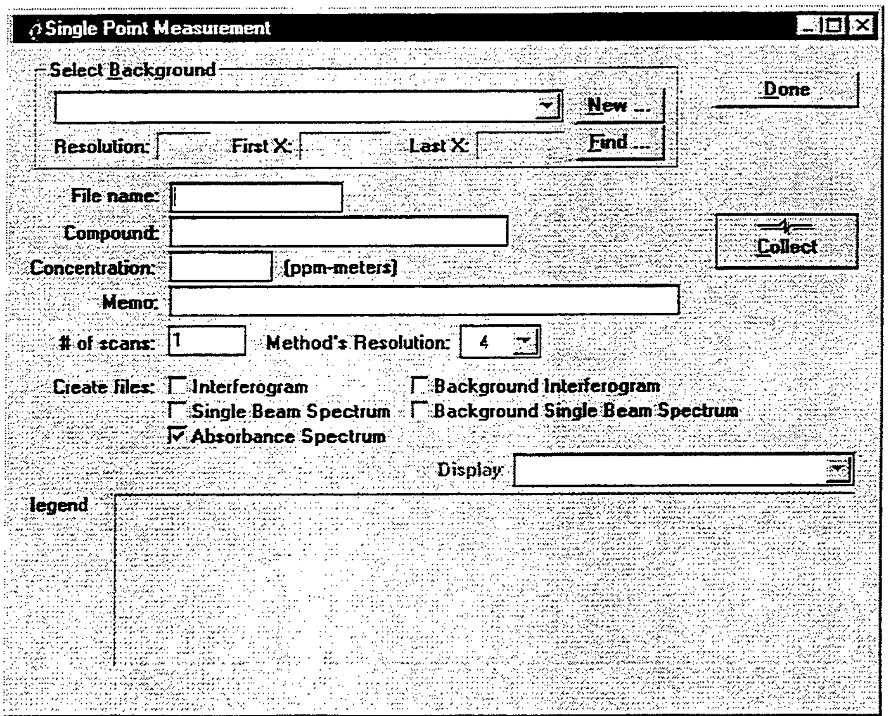

Figure V.A.2 The Single Point Measurement menu in AutoQuant™.

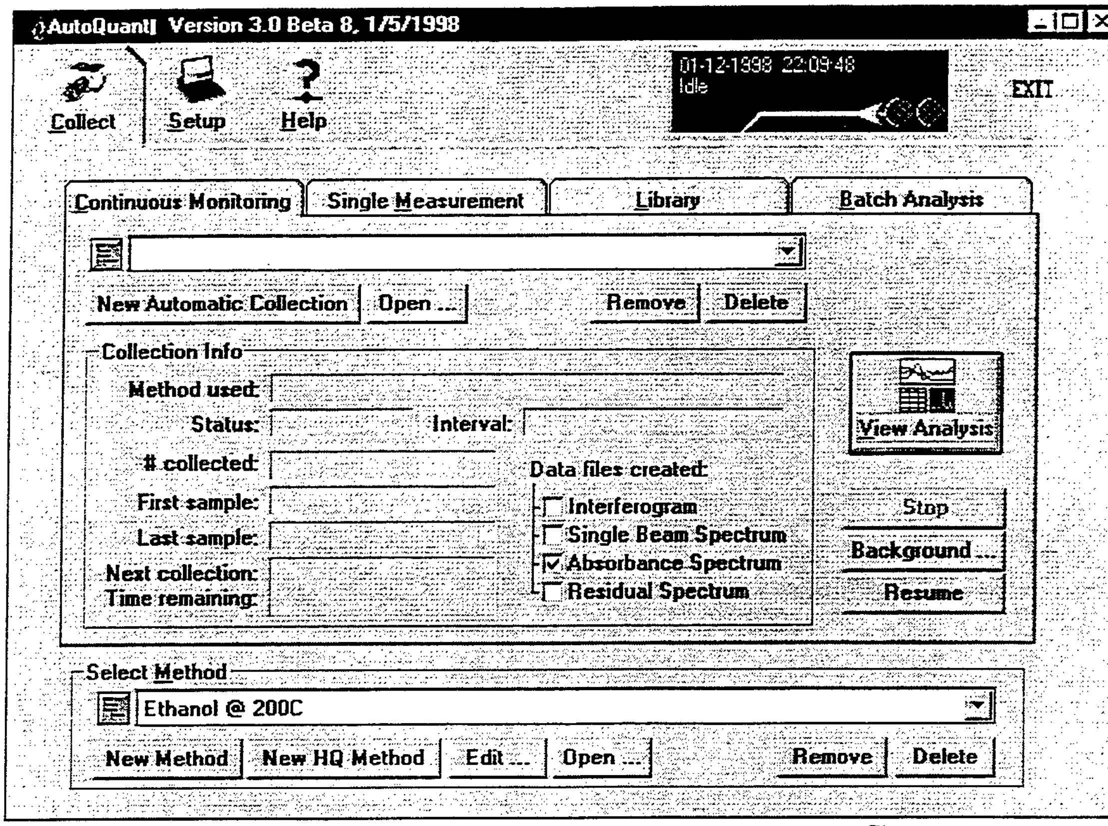

Figure VI.A.1 The main view of the Continuous Monitoring menu in AutoQuant™.

menu, click on the *View Analysis* button and the results of the collection will be displayed.

To create a new continuous collection, click on the *New Automatic Collection* button. This button will open the Continuous Monitoring: New Collection menu, shown in Figure VI.A.2. This menu allows the user to enter and set many important parameters, including:a meaningful description of the collection,the subdirectory in which spectra and results will be stored, a second computer path that will automatically back up the data collected, a memo that will be associated with all spectra collected during the experiment, the interval at which sample spectra will be collected,the number of samples that are to be collected, the number of coadded scans to be used for the data collection, the type of data files to be saved,the type of result files to be created,the background spectrum to be used for the collection, and finally,the analytical method to be employed.

Once all of this information is entered, the collection is initiated by clicking the *Start* button at the top right of the menu. This will lead to the Concentration vs. Time Graph Setup menu for the collection. This menu, shown in Figure VI.A.3, allows the user to choose the concentration and time units to be displayed in the results table and plot, the limits of the x and y axis of the plots and the components to be displayed on the plots.

Once the concentration vs. time information is entered, the collection is started by clicking on the *OK* button at the top right of the menu. Figure VI.A.4 shows the Analysis of Continuous Monitoring menu that is used to display the results of the continuous collection. The collection can be stopped from this menu in order to collect a new background or QA/QC data and then resumed once the user is finished.

The results of the collection are displayed in three ways. First, the concentration is plotted against time at the top of the screen. The y units of this plot can be changed to include temperature and pressure corrections if temperature and pressure sensors are present. Likewise, the format of the plot can be changed to display mass emissions if the system is connected to a mass flow controller.

The second way the results are displayed in this window is the table of concentration and time displayed in the lower left corner of the window. This table can be browsed as the collection progresses to see numerical values for the analysis results. Also, this table can be saved as a text file to be imported into a spreadsheet or database program.

The final way the data is displayed in this window is the spectrum viewer that is in the bottom right hand corner of the window. This viewer allows the user to visually inspect either the spectra that are being collected or the residual spectrum of the analysis.

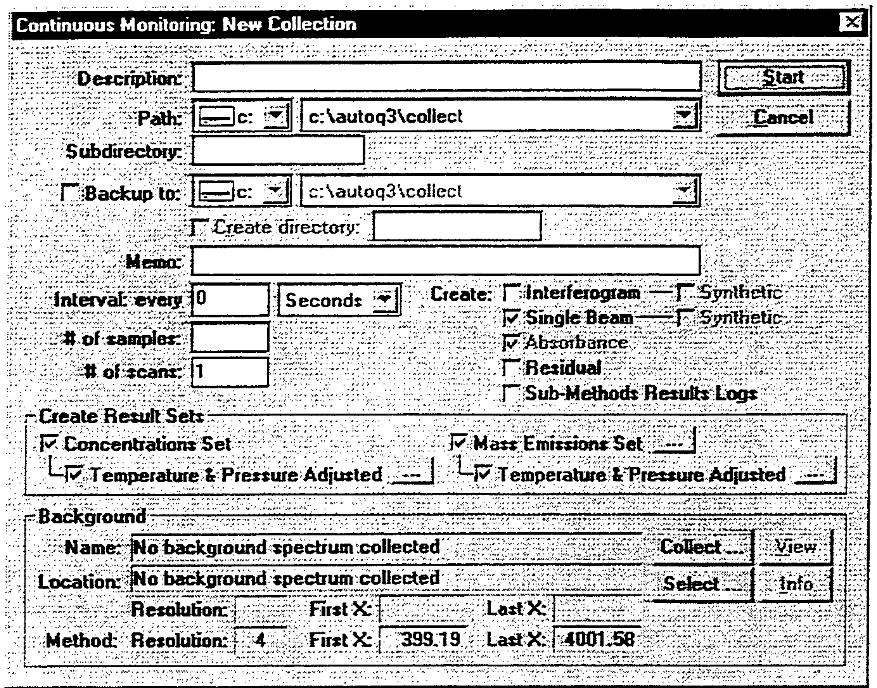

Figure VI.A.2 The New Collection menu for Continuous Monitoring in AutoQuant™.

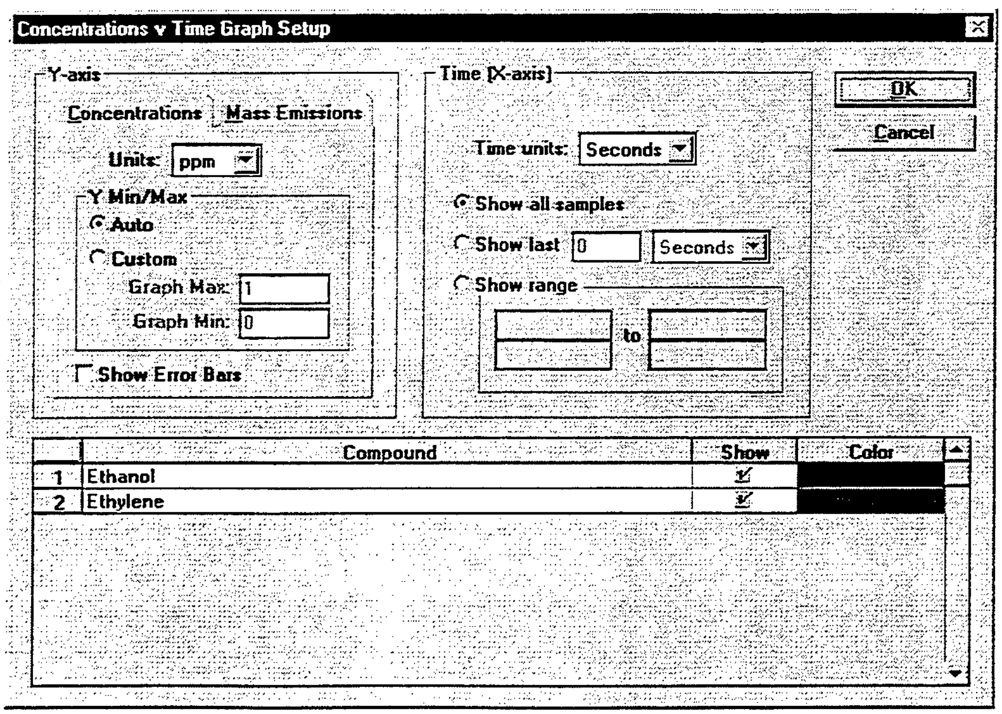

Figure VI.A.3 The Concentration vs. Time Graph Setup for continuous monitoring in AutoQuant™.

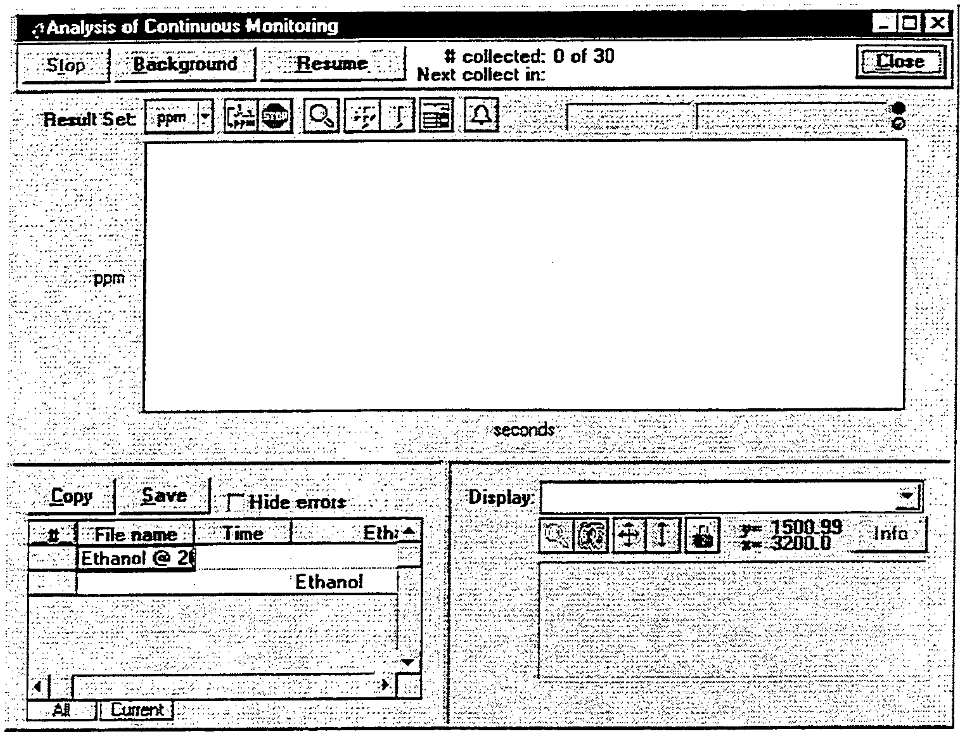

Figure VI.A.4 The Analysis of Continuous Monitoring view in AutoQuant™.

VII. Performing a Batch Analysis

The process of performing a batch analysis was covered in the earlier manual "Hardware, Software, and Operational Manuals for an Extractive FT-IR Air Monitoring System" that was delivered to the U.S. Army in November of 1997. Therefore, most of the contents of this section have been taken directly from that manual with only minor modifications to accommodate the new version of AutoQuant™.

Performing a Batch Analysis

The Batch Analysis window of the main menu in AutoQuant™ is shown in Figure VII.A.1. This menu allows

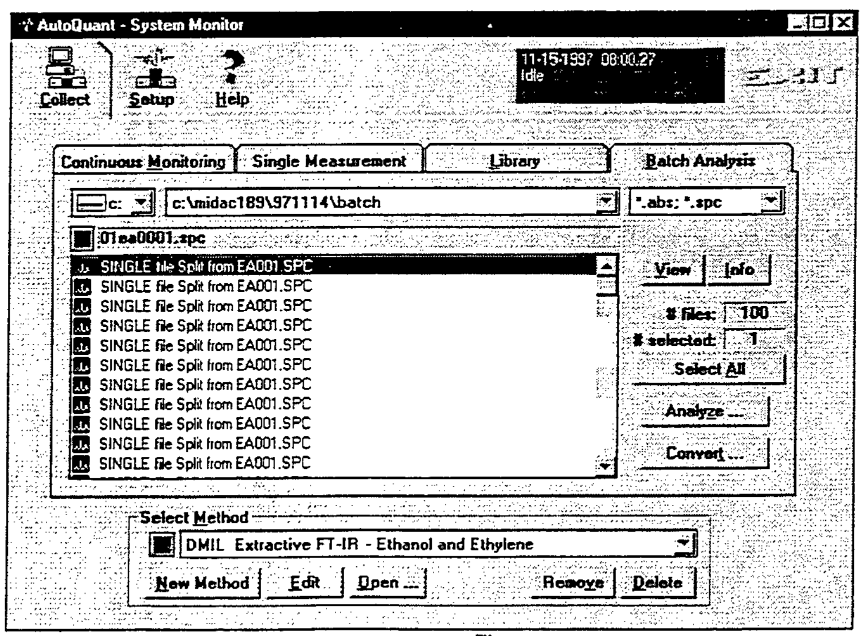

Figure VII.A.1 The Batch Analysis window in AutoQuant™.

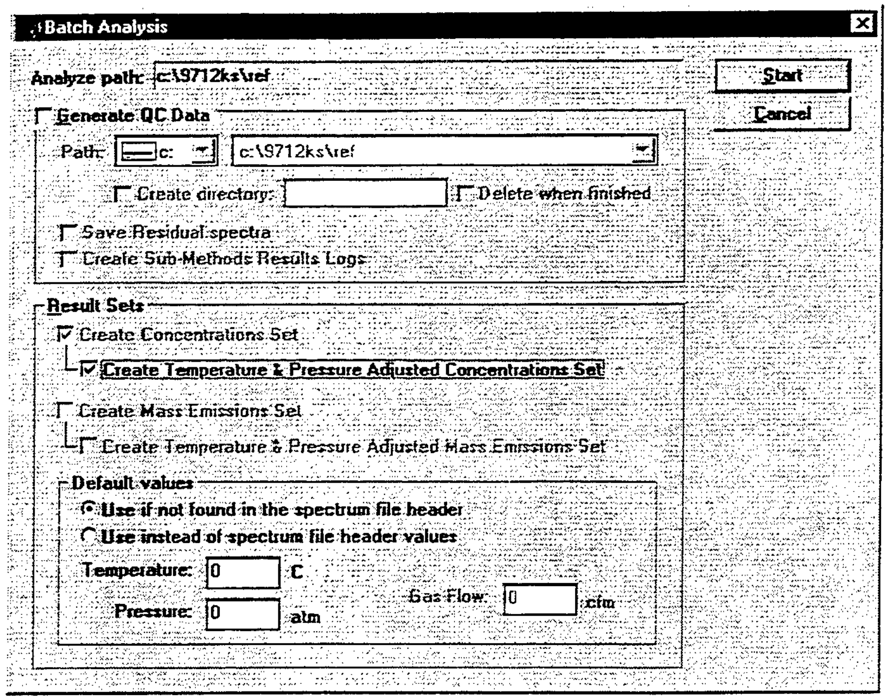

Figure VII.A.2 The Batch Analysis menu in AutoQuant™.

the user to select any or all spectra within a given directory for analysis by the analytical method that is shown at the bottom of the menu. Once the files to be analyzed are selected, clicking the Analyze button starts the batch analysis.

At this point, the Batch Analysis menu shown in Figure VII.A.2 appears. This menu determines if the residual spectrum is saved for viewing by the user. The residual spectrum is the spectrum that remains after the subtraction of the algorithm's suggested spectrum from the real spectrum. If the analysis is correct, this spectrum will be made up exclusively of noise in the analytical regions that were specified by the analytical method. This spectrum should be saved for viewing because it is the best tool available for the evaluation of the analysis results. The results of the different sub-methods that may be employed in a nonlinear analysis can also be saved from this menu.

In addition, temperature and pressure corrections can be generated from this menu. If the temperature and pressure of the sample are not present in the header of the samples, a temperature and pressure can be specified for the data at this menu. If the temperature and pressure are specified on this menu, then all spectra analyzed by the analytical method will be assigned the same temperature and pressure.

Once the appropriate information is input on this menu, clicking the Start button at the top right corner of the batch analysis menu starts the batch analysis.

Before the actual batch analysis begins, the user will prompted to enter the appropriate concentration vs. time information on the Concentration vs. Time Graph Setup menu shown in figure VII.A.3. When the appropriate information has been entered, the batch analysis is initiated by clicking the *OK* button at the top right corner.

Figure VII.A.4 is the *Batch Analysis Results* view. This view shows a plot of concentration versus time at the top, a table of results at the bottom left, and the spectrum display at the bottom right. There are two things to note here. First, the analysis is not performed in sequential order on the selected data. As the table of results shows, AutoQuant™ starts with a random spectrum out of the selected spectra. This can be a source of irritation when plotting the analysis results in a spreadsheet or database program. The second thing to note is that the concentration versus time plot does not always appear when performing a batch analysis. This is due to a bug in the programming. If the concentration vs. time plot does appear, the plot can be expanded or the concentration axis can be changed using the buttons at the top of the plot.

The residual spectrum should be viewed in the spectrum display to determine how well the analysis worked. Ideally, the error that is listed in the table of results would be the main indication of the quality of the results. However, because of the way that Midac AutoQuant™ performs the baseline correction and the subsequent subtraction of the analytical regions, this error is not always correct. Therefore, viewing the residual spectrum for indications of any analyte or interferant still present in the residual spectrum is the best evaluation of the batch analysis results.

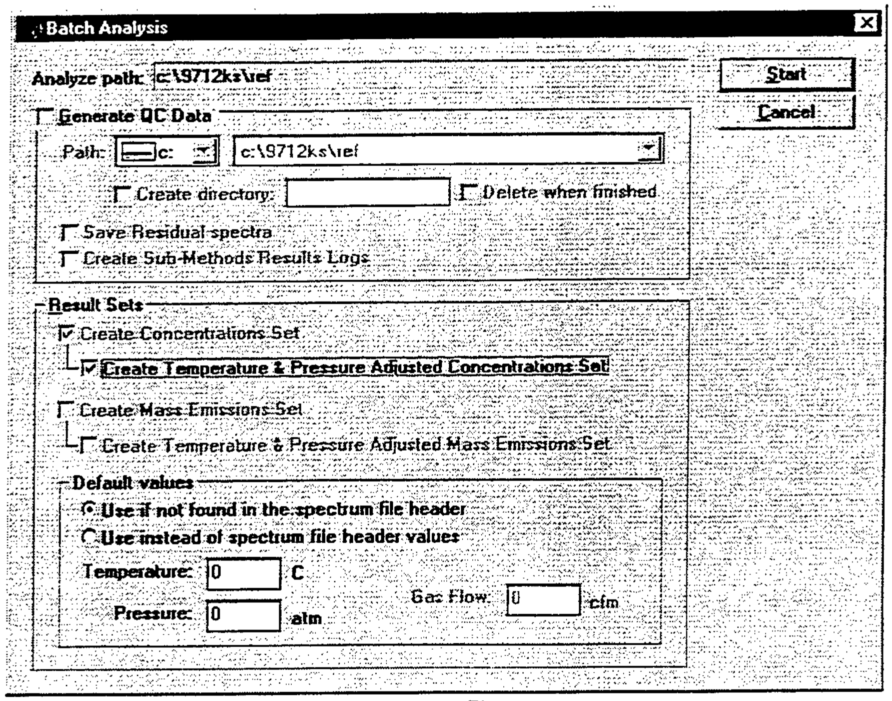

Figure VII.A.2 The Batch Analysis menu in AutoQuant™.

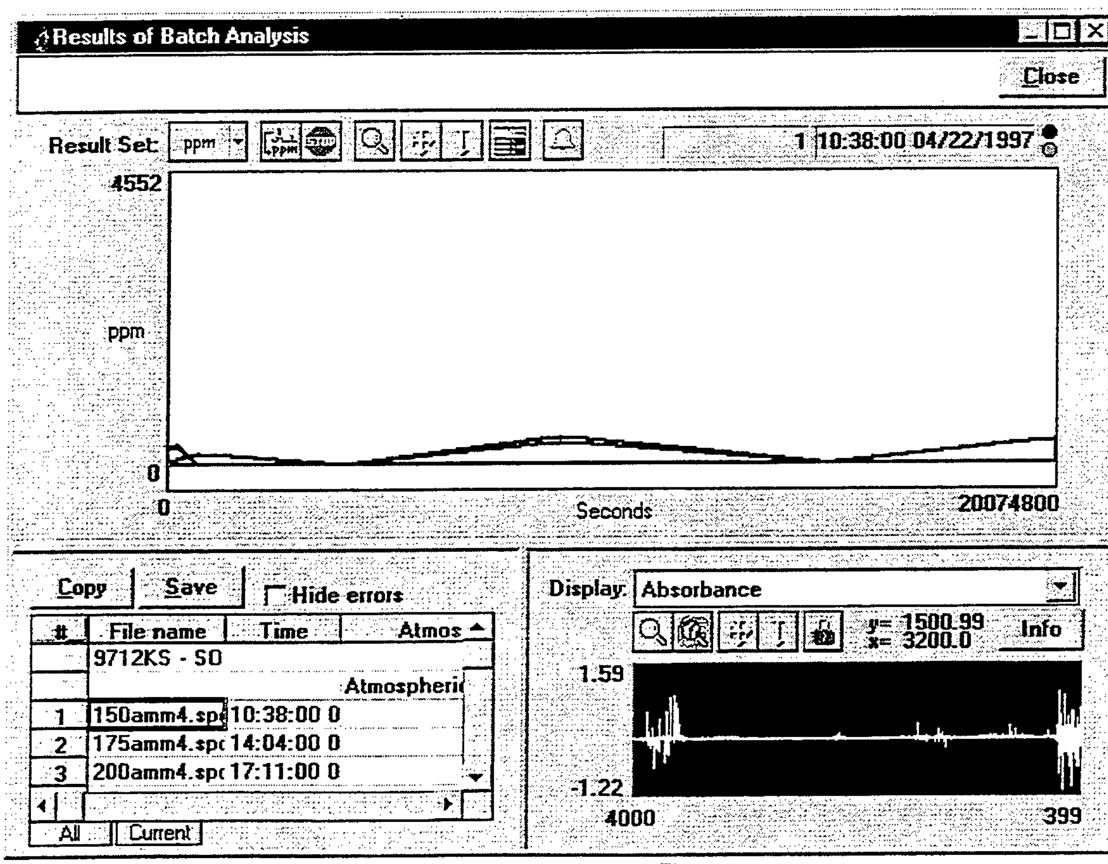

Figure VII.A.4 The Batch Analysis Results view in AutoQuant™.

Appendix A: Questions Concerning the Fourier Transform Routine within Midac AutoQuant™ Version 3.0

In November of 1997, a discrepancy was found between the FFT routines used in MDGrams/32™ and AutoQuant™. The discrepancy seemed to be present in spectra that displayed sharp absorption features, but not in spectra that displayed broad absorption features. The problem presented itself in two ways, both apparent in spectra processed using AutoQuant™.

First, there seemed to be a noise component added into the affected spectra, and second, the peak intensity of absorbance bands was noted to be almost half of that observed in MDGrams/32™. This phenomenon was observed repeatedly in November of 1997. Midac was informed of the problem and the raw data was sent to Midac for their evaluation.

Before this manual was undertaken in late December of 1997, an attempt was made to reproduce the anomalous results with the newest version of AutoQuant™. After the problem was first noted in November, Midac had suggested that the problem might involve mixing and matching apodization functions and zero filling of the collected interferograms. Data was collected using the same apodization function and zero filling parameters in the newest version of AutoQuant™ to determine if the problem was still present.

The data collected consisted of eight interferograms. Two background interferograms in AutoQuant™, two sample interferograms in AutoQuant™, two background interferograms in MDGrams/32™, and two sample interferograms in MDGrams/32™. In all cases, the background spectra collected were of pure nitrogen flowing through a 10-cm gas cell. Likewise, all sample spectra collected were of a 352.2-ppm*m ethylene certified reference gas.

After the data was collected, absorbance spectra of the samples were created using three separate processing methods. First, the interferograms collected in AutoQuant™ were processed using the FFT routine in AutoQuant™ using first a boxcar apodization and 2x zero filling and then a triangular apodization function and 2x zero-filling. Similarly the interferograms collected in MDGrams/32™ were processed using the FFT routine in MDGrams/32™ using first boxcar apodization and 2x zero-filling and then using triangular apodization and 2x zero-filling. Finally, the interferograms collected using AutoQuant™ were processed using the FFT routine in MDGrams/32™ using first a boxcar apodization and 2x zero filling and then a triangular apodization function and 2x zero-filling.

The results can be seen in Figures A1 and A2. Figure A1 shows the overlay of the samples processed using a boxcar apodization function and 2x zero filling. Figure A2 shows the overlay of the samples processed using a triangular apodization function and 2x zero filling.

As can be seen, the data collected with MDGrams/32™ displays a more intense absorption band than does either of the spectra collected in AutoQuant™, whether processed in AutoQuant™ or MDGrams/32™. The situation is worst using a boxcar apodization function. In this situation, not only is the spectrum collected in MDGrams/32™ more intense than the spectrum collected and processed in AutoQuant™, but the spectrum collected in AutoQuant™ and processed in MDGrams/32™ shows a large discrepancy when compared to the spectrum collected and processed in AutoQuant™. These spectra were created from the same interferograms.

No explanation for this problem is readily apparent. The only solution that can be suggested is that all sample spectra be analyzed by reference library spectra collected and processed in the same software package as the sample data. This is generally a good practice to follow whether errors such as this are present or not. However, it appears to be mandatory in this situation.

There is one good note from this experiment. The noise component that was observed in November of 1997 did not appear in this data. This may be an indication that it was an instrument problem instead of a software problem. This may never be proven, though, and users of AutoQuant™ should be vigilant in their periodic visual evaluation of all data collected.

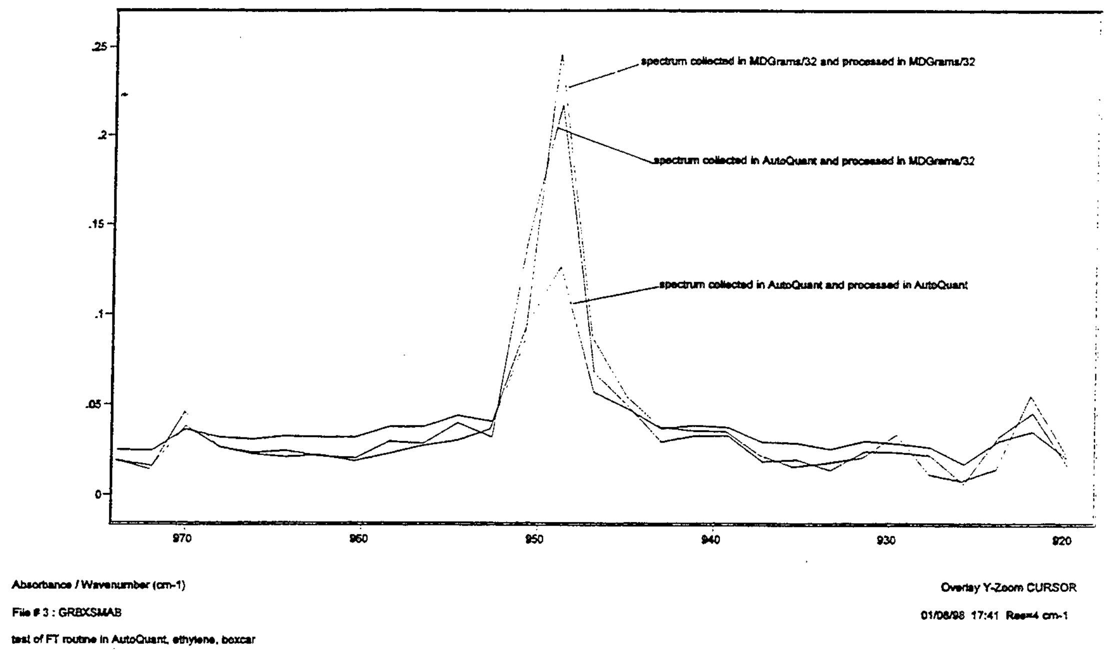

Figure A1. An overlay of the spectra processed using a boxcar apodization function and 2x zero-filling.

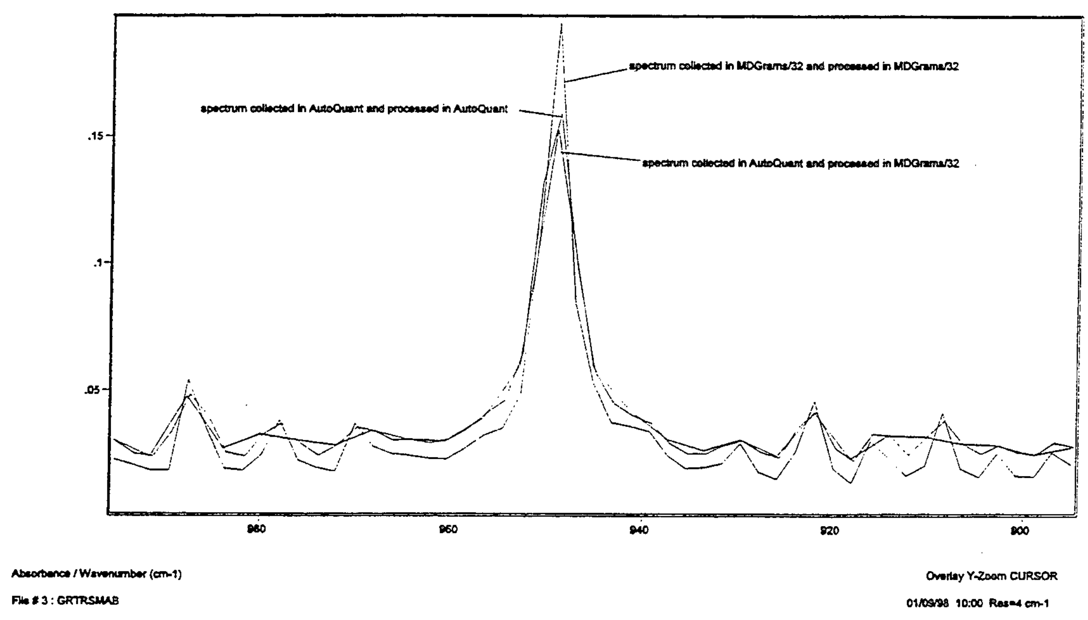

Figure A2 An overlay of the spectra processed using a triangular apodization function and 2x zero-filling.